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Indian Standard

IS 330 : 2023

क्रोमियम ट्राइऑक्साइड — विशिष्टि

(तीसरा पुनरीक्षण)

Chromium Trioxide — Specification

(Third Revision)

ICS 25.220.40

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Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

In the electroplating industry chromium trioxide (often called chromic acid) is used in making up the chromium plating bath used for decorative coating over copper or nickel; protective coatings on all basis metals; hard deposits over ferrous metals for tools, dies and gauges; for giving an oxide film on aluminium surface; porous coatings for improved lubrication, etc.

This standard was originally published in 1951. In the first revision of this standard, the chromium trioxide content was stipulated at 98.5 percent by mass as compared to that of 98 percent specified in the original standard. In addition, requirements of analytical grade material, previously covered in IS 559 : 1954 'Specification for chromium trioxide (chromium acid)', analytical reagent, were also included. In the second revision, special requirements for chromium trioxide for electronic industry had been added to make the standard more comprehensive.

In this revision alternative instrumental test method (AAS) for iron has been incorporated and the standard has been updated based on the experience of last three decades and on the currently available data.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CHROMIUM TRIOXIDE — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for chromium trioxide (chromic acid) for use in electroplating, anodizing work and as analytical reagent.

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
IS 264 : 2005	Nitric acid — Specification (*third revision*)
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 266 : 1993	Sulphuric acid — Specification (*third revision*)
IS 296 : 1986	Specification for sodium carbonate, anhydrous (*third revision*)
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 695 : 2020	Acetic acid — Specification (*fourth revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 1260 (Part 1) : 1973	Pictorical markings for handling of goods: Part 1 Dangerous goods (*first revision*)
IS 13320 : 1992	Determination of iron by atomic absorption spectrometry — Test method

3 GRADES

There shall be two grades of the material, as follows:

a) Grade I — Electroplating grade, suitable for electroplating and anodizing; and

b) Grade II — Analytical reagent grade.

4 REQUIREMENTS

4.1 Description

4.1.1 *Electroplating Grade*

The material shall consist of reddish to dark brownish granules of flakes of highly hygroscopic and corrosive character.

4.1.2 *Analytical Reagent Grade*

The material shall be in the form of dark, brownish-red crystals of granular powder of highly hygroscopic character.

4.2 Solubility

The material of electroplating grade shall be highly soluble in water; 10 g of it when dissolved in 25 ml of water shall give a clear red solution on settling. The material of analytical reagent grade shall be readily soluble in water, forming a clear, orange coloured solution.

4.3 The material shall also comply with the requirements laid down in Table 1, when tested in accordance with the method prescribed in Annex A.

For use in hard chrome plating, the requirements of chromium trioxide (as CrO_3), matter insoluble in water and sulphate (as SO_4) may be as agreed to between the purchaser and the manufacturer.

4.4 Special Requirements for Electronics Industry

For electronics industry, the material shall also comply with the requirements given in Table 2 in addition to those given in Table 1, when tested in accordance with the methods prescribed in Annex A.

5 PACKING

5.1 The material shall be packed in wide-mouth glass bottles or jars provided with well-ground air-tight glass stoppers. The electroplating grade material may also be packed in suitable air-tight steel drums.

The material, when packed, shall not come in contact with organic substances, as otherwise violent action might result.

CAUTION — Due to its corrosive and poisonous nature, avoid contact with skin.

Table 1 Requirements for Chromium Trioxide

(*Clauses* 4.3, 4.4 *and* 6.1.2)

Sl No.	Characteristic	Requirements for		Method of Test, Ref to
		Electroplating Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)
i)	Chromium trioxide (as CrO_3), percent by mass, *Min*	98.5	99.00	**A-2**
ii)	Matter insoluble in water, percent by mass, *Max*	0.15	To pass the test	**A-3**
iii)	Sulphates (as SO_4), percent by mass, *Max*	0.20	To pass the test	**A-4**
iv)	Alkali salts (as Na_2O), percent by mass, *Max*	0.30	0.200	**A-5**
v)	Chlorides (as Cl), percent by mass, *Max*	0.05	0.005	**A-6**
vi)	Nitrates (as NO_2), percent by mass, *Max*	—	0.004	**A-7**
vii)	Aluminium, iron and trivalent chromium (as R_2O_3), percent by mass, *Max*	—	0.030	**A-8**

Table 2 Additional Requirements for Chromium Trioxide for Electronics Industry

(*Clauses* 4.4 *and* 6.1.2)

Sl No.	Characteristics	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Sulphates (as SO_4), percent by mass, *Max*	0.005	**A-4.5**
ii)	Iron (as Fe), percent by mass, *Max*	0.01	**A-9**
iii)	Potassium (as K), percent by mass, *Max*	0.1	**A-10**
iv)	Sodium (as Na), percent by mass, *Max*	0.05	**A-11**

6 MARKING

6.1 The containers, and also the packages where possible, shall be suitably marked in red letters not less than 2.5 cm high; and drums shall be marked in red letters not less than 5 cm high, with the words '**CORROSIVE, HANDLE WITH CARE'**. They shall also be marked with the name, and grade of the material; its weight and indication of the source of manufacture.

6.1.1 The packages labelled as shown IS 1260 (Part 1).

6.1.2 *For Analytical Reagent Grade*

In addition, the containers of the analytical reagent grade material shall also be labelled with full analytical data for all the characteristics of that grade prescribed in Table 1 and Table 2.

7 SAMPLING

The method of preparing representative samples of the material and the criteria for conformity of the material to this specification shall be judges as prescribed in Annex B.

ANNEX A

(*Clause* 4.3)

ANALYSIS OF CHROMIUM TRIOXIDE

A-l QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF CHROMIUM TRIOXIDE

A-2.1 General

Chromium trioxide is determined by titrating the liberated iodine using starch solution as indicator.

A-2.2 Reagents

A-2.2.1 *Hydrochloric Acid*

Approximately 10 percent solution, prepared by diluting 270 ml of concentrated hydrochloric acid (*see* IS 265) of specific gravity l.16 with water to 1 000 ml.

A-2.2.2 *Potassium Iodide* — solid, free from iodate

A-2.2.3 *Standard Sodium Thiosulphate Solution*

Approximately 0.1 N. Dissolve about 25 g of sodium thiosulphate crystals ($Na_2S_2O_3.5H_20$) in freshly boiled distilled water, add 0.1 g of sodium carbonate (*see* IS 296) and make up the volume to 1 000 ml. Allow the solution to stand for a day and then siphon out the supernatant liquid. Pipette 25 ml of standard potassium dichromate solution (0.1 N) into a conical flask. Add 5 ml of hydrochloric acid, 2 g of potassium iodide and rotate to mix. Allow to stand for 5 min and then add 100 ml of water. Titrate with the thiosulphate solution, using starch solution as indicator towards the end.

A-2.2.4 *Starch Solution*

Triturate 1 g of pure soluble starch with 30 ml of water in a mortar. Pour the resulting paste into 1 litre of boiling water, boil for 3 min, allow the solution to cool and decant off the clear liquid. Prepare just before use.

A-2.3 Procedure

Accurately weigh, out of an air-tight weighing bottle, about 5 g of the material and dissolve it in water. Make the solution up to exactly 1 000 ml and mix thoroughly. Pipette out 25 ml into a glass-stoppered flask, add 5 ml of hydrochloric acid and 3 g of potassium iodide and let it stand in the dark for 15 min. Dilute with 100 ml of water, and titrate the liberated iodine with standard sodium thiosulphate solution, adding starch solution towards the end of the titration and noting the end-point when the blue colour just disappears. Carry out a blank test of the reagents immediately.

A-2.4 Calculation

Chromium trioxide (as CrO_3), percent by mass

$$= \frac{133.3 \times \mathrm{N} \times (V - V_1)}{M}$$

where

N = normality of the standard sodium thiosulphate solution used;

V = volume, in ml, of standard sodium thiosulphate solution required by 25 ml of the solution of the material;

V_1 = volume, in ml, of thiosulphate solution required in the blank test; and

M = mass, in g, of the material taken for the test.

A-3 DETERMINATION OF WATER INSOLUBLE MATTER

A-3.1 Procedure

Weigh accurately, out of an air-tight weighing bottle, about 20 g of the material and dissolve it in 200 ml of water. Heat on a steam bath for 1 h. Filter through a tared Gooch crucible with an asbestos mat or a sintered glass crucible (G No. 4). The asbestos mat on the Gooch crucible should be previously treated with a hot solution of chromic acid and dried before taring. Wash well and dry the crucible to constant weight at 105 °C to 110 °C. Cool the crucible in a desiccator and weigh.

A-3.2 Calculation

$$\text{Insoluble matter, percent by mass} = \frac{100\,M1}{M}$$

where

M_1 = mass, in g, of the residue; and

M = mass, in g, of the material taken for the test.

A-3.3 Determination of Insoluble Matter in Material of Analytical Reagent Grade

Weigh accurately, out of an air-tight weighing bottle, about 25 g of the material, dissolve it in water and dilute to 250 ml.

The material shall be taken to have passed the test if the solution obtained is clear and orange coloured.

A-4 DETERMINATION OF SULPHATES

A-4.1 General

Sulphate are determined in the material of electroplating grade by gravimetric method and, of analytical reagent grade and electroplating grade for electronics industry by turbidity test.

A-4.2 Reagents

A-4.2.1 *Rectified Spirit* — 95 percent by volume (*see* IS 323)

A-4.2.2 *Concentrated Hydrochloric Acid* — sp gr 1.16 (*see* IS 265)

A-4.2.3 *Barium Chloride Solution* — approximately 10 percent (*m/v*)

A-4.2.4 *Dilute Hydrochloric Acid* — approximately one percent (*v/v*)

A-4.2.5 *Acetic Acid* — glacial

A-4.3 Procedure

Weigh accurately about 10 g of the material and dissolve in 50 ml of water. Filter into a glass beaker through a 7 cm filter paper to eliminate any insoluble matter. Wash the filter paper well with water and add to the filtrate 10 ml of rectified spirit, 35 ml of concentrated hydrochloric acid and 10 ml of glacial acetic acid. Heat until all the chromate is reduced to the green trivalent form. Boil off most of the aldehyde formed, and add to the solution 6 ml to 8 ml of barium chloride solution through the same filter paper. Dilute the solution to 150 ml with water, heat the solution without boiling for 4 h and allow to stand overnight. Filter the solution containing barium sulphate through a tared sintered glass crucible (G No. 4) or a Gooch crucible. Wash the barium sulphate residue with dilute hydrochloric acid till the residue is free from chromium salts and then with water till it is free from chlorides. Dry the residue to constant weight at 105 °C to 110 °C, and weigh. Carry out a blank test on reagents immediately.

NOTES

1 After standing overnight filter again if there is any precipitate.

2 Excess of barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution-by addition of barium chloride in a slow stream with Stirring minimizes mechanical occlusion of barium chloride and gives a coarse precipitate which is less soluble in acids.

A-4.4 Calculation

$$\text{Sulphates (as } SO_4)_3\text{, percent by mass} = \frac{41.15 \times M_1}{M}$$

where

M_1 = mass, in g, of the barium sulphate residue; and

M = mass, in g, of the material taken for the test.

A-4.5 Turbidity Test for Sulphates in Material of Analytical Reagent Grade

A-4.5.1 *Reagents*

A-4.5.1.1 *Dilute hydrochloric acid* — approximately 5 N

A-4.5.1.2 *Barium chloride solution* — approximately 12 percent (*m/v*)

A-4.5.2 *Procedure*

Dissolve 4 g of the material in 50 ml of water, add 1 ml of dilute hydrochloric acid and 1 ml of barium chloride solution. Allow to stand for 3 h.

The material shall be taken to have satisfied the requirement of the test if no turbidity or a precipitate is produced in 3 h.

A-4.6 Turbidity Test for Sulphates in Material of Analytical Reagent Grade

A-4.6.1 *General*

Sulphates are determined by comparing the turbidity produced by the material with barium chloride solution against that produced by a standard sulphate solution.

A-4.6.2 *Reagents*

A-4.6.1.1 *Dilute hydrochloric acid* — approximately 5 N

A-4.6.1.2 *Barium chloride solution* — approximately 10 percent

A-4.6.1.3 *Standard sulphate solution*

Dissolve 0.181 4 g of potassium sulphate in water

and make up the volume to 1 000 ml. Take 10 ml of this solution and dilute it further to 100 ml. [1 ml of the final solution = 0.01 mg of sulphate (as SO_4)]

A-4.6.2 *Procedure*

Dissolve 4 g of the material, accurately weighed in 50 ml of water in a Nessler Cylinder. To this solution, add 1 ml of dilute hydrochloride acid and 2 ml of barium chloride solution. Stir the mixture and allow to stand for 20 min. Carry out a control test in the other Nessler cylinder using 2 ml of standard sulphate solution and the same quantities of other reagents, finally diluting to the mark A.

The limit shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-5 DETERMINATION OF ALKALI SALTS

A-5.1 Procedure

Carefully ignite in a porcelain dish, about 1.5 g of the material accurately weighed out of an air-tight weighing bottle, and triturate with 10 ml of hot water. Filter through a small filter paper and wash with 10 ml of hot water. Evaporate the extract. Warm the residue with 30 ml of water, filter, wash with 10 ml of hot water and evaporate in the same dish. Ignite the evaporated residue to constant weight.

A-5.2 Calculation

$$\text{Alkali salts } (Na_2O)_3\text{, percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = mass, in g, of the residue; and
M = mass, in g, of the material taken for the test.

A-6 DETERMINATION OF CHLORIDES

A-6.1 General

Chlorides are determined in the material of electroplating grade by gravimetric method and, of analytical reagent grade by turbidity test.

A-6.2 Reagents

A-6.2.1 *Concentrated Nitric Acid* — sp gr 1.42 (*see* IS 264)

A-6.2.2 *Silver Nitrate Solution* — approximately 5 percent (m/v)

A-6.2.3 *Dilute Ammonium Hydroxide Solution* — approximately 4 N

A-6.2.4 *Dilute Nitric Acid* — approximately 4 N

A-6.3 Procedure

Weigh accurately about 10 g of the material and dissolve in 150 ml of water. Filter to obtain a clear solution. Wash the filter paper carefully and collect the washing in the beaker containing the filtrate. Make up the resulting solution to about 200 ml. Add a few drops of concentrated nitric acid and then an excess of silver nitrate solution. Allow the solution to stand overnight. Filter the solution through a Gooch crucible or sintered glass crucible (G No. 4). Wash the precipitate, first with dilute nitric acid to dissolve any adhering silver chromate and then with cold water. Dissolve the precipitate in dilute ammonium hydroxide solution and reprecipitate by the addition of dilute nitric acid. Filter through a tared Gooch crucible or sintered glass crucible (G No. 4) and wash with water. Dry the crucible and its contents to constant weight at (130 ± 2) °C.

A-6.4 Calculation

$$\text{Chlorides (as Cl), percent by mass} = \frac{24.74\ M1}{M}$$

where

M_1 = mass, in g, of silver chloride; and
M = mass, in g, of the material taken for the test.

A-6.5 Turbidity Test for Chlorides in Material of Analytical Reagent Grade

A-6.5.1 Reagents

A-6.5.l.l *Dilute nitric acid* — approximately 5 N

A-6.5.1.2 *Silver nitrate solution* — approximately 5 percent (*m/v*)

A-6.5.2 *Procedure*

Dissolve 1.000 g of the material in 20 ml of water and add 10 ml of dilute nitric acid. Heat the solution to 50 °C and add 1 ml of silver nitrate solution.

A-6.5.2.1 The material shall be taken to have not exceeded the limit prescribed in Table 1 if no turbidity is produced within 2 min.

A-7 TEST FOR NITRATES

A-7.1 Reagents

A-7.1.1 *Dilute Sulphuric Acid* — approximately 5 N

A-7.1.2 *Hydrochloric Acid* — approximately 11 N

A-7.1.3 *Dilute Ammonium Hydroxide Solution* — approximately 5 N

A-7.1.4 *Barium Chloride Solution*

Dissolve 3 g of barium chloride crystals in 10 ml of water and filter.

A-7.1.5 *Standard Potassium Nitrate Solution*

Dissolve 1.63 g of potassium nitrate in water and make up the volume to 1 000 ml. Take 10 ml of this solution and dilute again to 1 000 ml immediately before use; 1 ml of this solution is equivalent to 0.01 mg of nitrate (as NO_3).

A-7.1.6 *Standard Indigo Carmine Solution*

Dissolve 0.20 g of indigo carmine in 500 ml of dilute sulphuric acid, add 20 ml of hydrochloric acid and sufficient dilute sulphuric acid to produce 1 000 ml. Standardize the Solution so that 10 ml added to 2.0 ml of standard potassium nitrate solution is just decolourized on adding 12 ml of concentrated sulphuric acid and heating to boiling. [1 ml of the solution = 0.02 mg of nitrate (NO_3)].

A-7.1.7 *Concentrated Sulphuric Acid* — sp gr 1.84 (*see* IS 266)

A-7.2 Procedure

Weigh accurately 1 g of the material and dissolve in 5 ml of water. Add 6 ml of dilute ammonium hydroxide solution, heat to boiling, add barium chloride solution and filter. To 10 ml of the filtrate, add 1 ml of standard indigo carmine solution and 10 ml sulphuric acid and heat to boiling.

The limit prescribed in Table 1 shall be taken as not having been exceeded if the blue colour does not entirely disappear.

A-8 DETERMINATION OF ALUMINIUM, IRON AND TRIVALENT CHROMIUM

A-8.1 Reagents

A-8.1.1 *Ammonium Chloride* — solid

A-8.1.2 *Ammonium Hydroxide Solution* — sp gr 0.90

A-8.1.3 *Ammonium Nitrate Solution* — approximately 1 percent (m/v)

A-8.1.4 *Dilute Hydrochloric Acid* — 1 : l (v/v)

A-8.1.5 *Methyl Red Indicator Solution* — prepared by dissolving 0.15 g in 500 ml of water

A-8.2 Procedure

Weigh accurately about 5 g of the material and dissolve in 100 ml of water. Heat to boiling, add 1 g of ammonium chloride, continue boiling till the solution is clear and then add ammonium hydroxide solution till the solution is distinctly alkaline to methyl red solution. Boil off excess of ammonia and filter through a filter paper (Whatman No. 31 or 41). After washing the precipitate on the filter paper several times with ammonium nitrate solution, dissolve it in minimum quantity of dilute hydrochloric acid, add 1 g of ammonium chloride and repeat the precipitation as above. Boil the liquor with the precipitate and filter it through a filter paper (Whatman No. 31 or 41), wash the precipitate thoroughly with ammonium nitrate solution and then with water till the washings are free from chlorides. Ignite the precipitate in a tared crucible to constant weight.

A-8.3 Calculation

Aluminium, iron and trivalentchromium

$$(R_2O_3), \text{ percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = mass, in g, of the residue; and
M = mass, in g, of the material taken for the test.

A-9 DETERMINATION OF IRON (Fe)

A-9.1 General

Two methods for the determination of iron are described. Either of these may be used for general routine purposes, but in case of a dispute Method 1 shall be referee method.

A-9.2 Method 1

A-9.2.1 Reagents

A-9.2.1.1 *Concentrated nitric acid — see* IS 264

A-9.2.1.2 *Ammonium persulphate* — solid

A-9.2.1.3 *Butanolic potassium thiocyanate*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient n-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-9.2.1.4 *Dilute sulphuric acid* — approximately 10 percent (*v/v*)

A-9.2.1.5 *Standard iron solution*

Weigh 0.702 g of ferrous ammonium sulphate [$FeSO4\ (NH_4)_2.6H_2O$] and dissolve in 10 ml of dilute sulphuric acid.

Dilute with water to make up the volume to 1 000 ml. Transfer 10 ml of this solution to a 100 ml volumetric flask and again dilute with water to make up the volume to 100 ml. (1 ml of this solution = 0.01 mg of iron).

A-9.2.2 Procedure

Take about 0.5 g of the sample accurately, weighed, dissolve it in 10 ml water. Add 1 ml of concentrated nitric acid and boil. Cool, transfer the solution to a Nessler cylinder and add 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake for 30 s and allow the liquid to separate. Carry out a control test in the other Nessler cylinder, adding slowly from a burette a quantity of the standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture until the colours of butanol layers in the two cylinders are exactly matched.

A-9.2.3 Calculation

$$\text{Iron (as Fe), percent by mass} = \frac{V}{M} \times 0.001$$

where

V = volume, in ml, of standard iron solution required in the control test; and

M = mass, in g, of the material present in the aliquot of prepared sample solution.

A-9.3 Method 2

Alternatively iron can be determined by Atomic Absorption Spectrophotometer (AAS); follow the method as prescribed in IS 13320.

A-10 DETERMINATION OF POTASSIUM (K)

A-10.1 Reagents

A-10.1.1 *Rectified Spirit — see* IS 323

A-10.1.2 *Perchloric Acid* — 40 percent to 60 percent

A-10.1.3 *Concentrated Hydrochloric Acid — see* IS 265

A-10.1.4 *Phenolphthalein Indicator Solution*

Dissolve 0.1 g of the material in 100 ml of mine this factor, taking only small quantities 60 percent rectified spirit.

A-10.1.5 *Acetic Acid* — glacial (*see* IS 695) and 20 percent solution

A-10.1.6 *Sodium Cobaltinitrite Solution*

Dissolve 50 g of sodium cobaltinitrite and 25 g of sodium acetate in 250 ml of water. Allow the solution to stand for 24 h and then filter.

A-10.2 Procedure

A-10.2.1. Dissolve about 5 g of the material in 10 ml of water. Add 0.5 ml of 20 percent acetic acid and drop by drop, while stirring, 5 ml of the sodium cobaltinitrite solution and allow to stand for several hours, preferably overnight. Then filter through a filter crucible, previously washed with cold water containing a little reagent and dried at 105 °C ± 2 °C to constant mass and weighed to the nearest 0.000 2 g.

A-10.2.2 Wash the residue on the filter three times with 20 percent acetic acid using 5 ml each time, and twice with rectified spirit, using. 5 ml each time, dry at a temperature of 105 °C ± 2 °C to constant mass, cool in a desiccator and weigh to the nearest 0.000 2 g. One gram of a precipitate of the composition $K_2NaCo(No_2)_6$ H_2O is assumed to contain 0.172 2 g of potassium. It is preferable to determine this factor, taking only small quantities of a potassium salt.

A-10.3 Calculation

Potassium (as K), percent by mass

$$= \frac{M_1 \times 0.172\ 2 \times 100}{M}$$

where

M_1 = mass, in g, of the dried residue; and

M = mass, in g, of the material taken for the test.

A-10.4 Alternatively determine potassium by flame photometer at 766.5 mμ according to the directions of the manufacturer of the apparatus.

A-11 DETERMINATION OF SODIUM (Na)

A-11.1 Determine sodium by flame photometer at 589 m according to directions of the manufacturer of the apparatus.

ANNEX B

(*Clause* 7)

SAMPLING OF CHROMIUM TRIOXIDE

B-l GENERAL REQUIREMENTS OF SAMPLING

B-1.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

B-l.2 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-l.3 The sampling instrument shall be clean and dry.

B-l.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-l.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-l.6 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

B-l.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-l.8 Each sample containers shall be sealed air-tight after filling, and marked with full details of sampling, and the date of sampling.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacturer, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be as given below:

Sl No.	*Lot Size* (*N*)	*Sample Size* (*n*)
(1)	(2)	(3)
i)	3 to 15	3
ii)	16 to 40	4
iii)	41 to 110	5
iv)	111 to 180	6
v)	181 to 300	7
vi)	301 to 500	8
vii)	501 to 800	9
viii)	801 and above	10

B-2.3 In order to ensure randomness of selection, random number tables shall be used. If such tables are not available, the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2, 3 ,.....,..., up to r so on and where r is the integral part of N/n (*see* **B-2.2**). Every r^{th} container thus counted shall be withdrawn to constitute the sample.

B-3 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLES

B-3.1 From each of the containers selected according to **B-2.3**, a representative portion of the material sufficient for carrying out the tests, specified under **4**, shall be drawn and this shall constitute the individual sample.

B-3.2 From each of the individual samples, a small but equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample.

B-3.3 The material constituting the individual and composite samples shall be transferred to separate bottles and labelled with full identification particulars of the samples.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of chromium trioxide content shall be carried out on each of the individual samples.

B-4.2 Tests for the remaining characteristics shall be done on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

For chromium trioxide content, the mean and range of test results shall be computed as follows:

$$\text{Mean } (X) = \frac{\text{sum of individual test results}}{\text{number of tests}}$$

Range (R) = difference between the maximum and the minimum values of the test results.

The lot shall be declared to have satisfied the requirements for chromium trioxide content if $X - 0.6\,R > 98.5$ for electroplating grade and $x - 0.6\,r > 99.00$ for analytical reagent grade and for electroplating grade for electronics industry respectively.

B-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all other characteristics (of both the grades), the test results on the composite sample shall meet the corresponding specified requirements.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

Organization	*Representative(s)*
CSIR - Central Electrochemical Research Institute, Karaikudi	DR B. SUBRAMANIAN **(*Chairperson*)**
Bhabha Atomic Research Centre, Hyderabad	SHRI M. RAMANAMURTHY
Bharat Electronics Limited, Bengaluru	SHRIMATI PADMA SHARMA SHRI SHREEDHAR NADIGER (*Alternate* I) SHRIMATI ATHILAKSHMI (*Alternate* II)
Bharat Heavy Electrical Limited, New Delhi	REPRESENTATIVE
Brite Platers & Electrical Engineers, Bangalore	SHRI D. P. BHATTACHARYA
CMP Private Limited, Mumbai	SHRI D. T. THAKUR SHRI R. K. CHUG (*Alternate*)
Consolidated Metal Finishing Private Limited, New Delhi	SHRI SUNIL KUMAR SHRI P. GURUMOORTHI (*Alternate*)
Defence Research and Development Organization, Research Centre Imarat, Hyderabad	REPRESENTATIVE
Department of Defence Production, Controllerate of Quality Assurance (Materials), Kanpur	SHRI M. V. KUMBHALKAR SHRI RAMALALA (*Alternate*)
Department of Industrial Policy & Promotion, New Delhi	REPRESENTATIVE
Electroplating and Surface Finishing of Metals Division	DR G. N. K. RAMESH BABU DR N. RAJASEKARAN (*Alternate*)
Films Division, Ministry of Information and Broadcasting, Mumbai	REPRESENTATIVE
Gujarat Alkali and Chemicals Ltd., Baroda	REPRESENTATIVE
Hindustan Photo Films Manufacturing Company Limited, Ootacamund	SHRI P. JAGADEESWARAN SHRI B. B. CHAUDHARY(*Alternate*)
Indian Centre for Plastics in the Environment, Mumbai	REPRESENTATIVE
Indian Space Research Organisation - Space Applications Centre, Ahmedabad	SHRI JAYANT SHRI RAJENDRA N. GAIKWAD (*Alternate*)
Kongovi Electronics Private Limited, Bengaluru	REPRESENTATIVE
Liberty Chemical Works, Mumbai	REPRESENTATIVE
MSME Testing Center, New Delhi	SHRI IMRAN MUJAWAR SHRI VIPUL GAIKWAD (*Alternate*)
Metal Finishers Association of India, Mumbai	SHRI T. K. MOHAN
Ministry of Commerce and Industry, Department for Promotion of Industry and Internal Trade, New Delhi	REPRESENTATIVE
Ministry of Information and Broadcasting, New Delhi	REPRESENTATIVE
Office of Development Commissioner (MSME), New Delhi	DR N. KARTHIKEYAN
Platewell Process and Chemicals Limited, Vadodara	REPRESENTATIVE
Prasad Film Lab, Bengaluru	REPRESENTATIVE

Organization	*Representative(s)*
Ronuk Industries Limited, Mumbai	REPRESENTATIVE
Satyadev Chemicals Private Limited, Vadodara	REPRESENTATIVE
Security Printing and Minting Corporation of India Limited, New Delhi	SHRI S. N. LAHIRI SHRI P. MEHROTRA (*Alternate*)
Society for Advancement of Electrochemical Science and Technology, Karaikudi	REPRESENTATIVE
Space Application Centre ISRO, Ahmedabad	SHRI JAYANT SHRI SHARAD SHUKLA (*Alternate*)
Standard Alkali (Chemical Division), Navi Mumbai	REPRESENTATIVE
BIS Directorate General	A. K. LAL SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (CHEMICAL [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
MS BUNTY MUDI
SCIENTIST 'B'/ASSISTANT DIRECTOR
CHEMICAL, BIS

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